

2<sup>nd</sup> Interdisciplinary Annual PhD Conference on Material Science and Innovative Technologies InterMST 2024

**Book of Abstracts** 

Krakow, 4-5 March 2024



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# Welcome to the 2<sup>nd</sup> InterMST 2024

Dear PhD Conference Participants,

On behalf of the Scientific Committee and the Organizing Committee we are pleased to welcome you on the InterMST 2024 conference – Interdisciplinary Annual PhD Conference on Material Science and Innovative Technologies hosted on-line by the Łukasiewicz Research Network - Krakow Institute of Technology, 04-05 March.

The conference is devoted to exploring the richness of approaches, methodologies, and themes of the discipline in order to showcase a wide range of studies and provide a picture of the current state of research in the field of material science and innovative technologies. The interdisciplinary approach of the conference highlight the trajectories of the various scientific disciplines which allow for a progress in material science and innovative technologies – from the engineering and exact sciences to the natural sciences and medical disciplines.

"Book of Abstracts" comprises 25 extended abstracts that have been carefully selected on the basis of a peer review process. It includes state of the art in scientific considerations related to innovative materials and material characterization, advances in casting technology, high temperature and high entropy materials, advances in coatings technologies and finally additive technologies and advances in biomedical and optical technologies.

On behalf of the conference hosts, we would like to express our gratitude to the members of the Scientific Committee, the members of the Organizing Committee, and all the Authors for their effort and willingness to take part in the InterMST 2024 conference – Interdisciplinary Annual PhD Conference on Material Science and Innovative Technologies.

Yours faithfully, dr hab. Katarzyna M. Marzec dr hab. inż. Tomasz Dudziak mgr inż. Joanna Białoń

Krakow, March 2024

# General information

This conference is devoted to exploring the richness of approaches, methodologies, and themes of the discipline in order to showcase a wide range of studies and provide a picture of the current state of research in the field of material science, engineering and innovative technologies. The interdisciplinary approach of the conference highlight the trajectories of the various scientific disciplines which allow for a progress in material science and innovative technologies - from the engineering and exact sciences to the natural sciences and medical disciplines. The conference is addressed to PhD student.

The conference topics include:

- Modern materials for harsh conditions (corrosion degradation)
- Coatings (thermal spray coatings, CVD, PVD, slurry coatings and others)
- Mechanical properties (creep, tensile, bending, micro bending, micro scale tensile and others properties)
- Heat treatment (various processes inducing higher performance of materials)
- Computing simulations (Cellular automata (CA), neural network modelling (NNM) and others)
- Additive manufacturing (SLM, SLS and others)
- Non-destructive techniques (tomography, XRD scanning and others)
- Microstructure characterisation (SEM, SEM-FEG, TEM, STEM and others)
- Tribology and wear properties
- High temperature ceramics (borides, oxides, carbides)
- Others related to Material Science and Engineering.

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# Abstracts

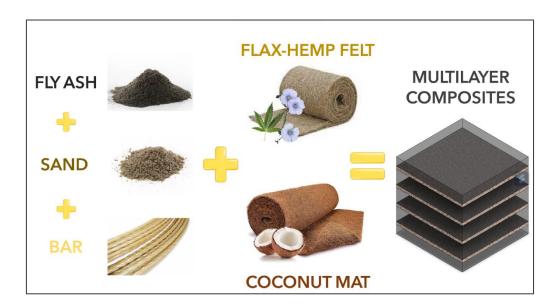
## INFLUENCE OF THE ADDITION A NATURAL FIBERS ON THE INSULATING PROPERTIES OF MULTILAYER GEOPOLYMER COMPOSITES

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In recent years, there has been a trend of rising material prices in the construction sector. The biggest jump has been among materials for insulating buildings. Numerous attempts and projects are being undertaken around the world to develop low-carbon materials, as well as advanced thermal insulation materials. The present work is related to the study of the insulating properties of innovative geopolymer composites with the addition of natural fibers for the production of prefabricated layered building envelopes. Natural fibers of plant origin used in the production of samples are biodegradable and primarily renewable materials. In the present study, flax felt and coconut mats, commonly used for mattresses, were used. A 10-mol sodium hydroxide solution with an aqueous solution of sodium silicate was used for the alkaline activation of geopolymers. The paper presents the results of physicochemical studies of fly ash from the Skawina Power Plant and river sand from the Świętochłowice Sand Plant. The physical and thermal parameters of the finished geopolymer multilayer partitions were also studied. The results confirm the high potential of these materials as a finished prefabricated product. The above material can be successfully used in the low-carbon, ecological, and energy-efficient construction sector.



Graphical abstract

#### MAGNESIUM-BASED BIOMATERIAL

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Resorbable biomaterials based on magnesium and it's alloys have emerged as promising candidates for medical applications, particularly in bone implants, owing to their unique combination of mechanical properties, biocompatibility, and degradability [1, 2]. These materials offer a solution to the limitations of permanent implants by providing temporary support for bone healing and regeneration while gradually degrading in the body.

This abstract presents a research in the field of resorbable magnesium-based biomaterials for bone implants. It discusses fabrication techniques, including alloy composition and processing methods, aimed at optimizing mechanical strength and degradation rates to match the healing process of bone tissue. We are researching the biological response to these implants, focusing on functional use.

The abstract also addresses challenges such as controlling degradation kinetics and ensuring adequate mechanical stability during the healing period. Overall, resorbable biomaterials based on magnesium and its alloys hold significant promise for advancing bone implant technologies, offering improved patient outcomes and reducing the need for secondary surgeries associated with permanent implants.

[1] J. Marciniak, Biomateriały, Wyd. Politechniki Śląskiej, Gliwice 2013, ISBN 978-83-7880-062-0

[2] A. Dudek, Rodzaje modyfikacji powierzchni biomateriałów stosowanych w medycynie, Wyd. PK, Kraków 2022, ISBN 978-83-67188-20-3

#### Mo-Si-B ALLOYS AS ULTRA-HIGH MATERIALS BEYOND SUPERALLOYS: FROM SESSILE DROP EXPERIMENTS TO HOT-DIPPING FABRICATION

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Boron doped Molybdenum Silicides (Mo-Si-B alloys) have been recognized as particularly attractive candidates for ultra-high temperature applications (both as a bulk components or coatings) due to a very good high mechanical strength, low specific weight and good oxidation resistance at temperatures beyond limits of conventional metallic heat resistant alloys. However, in order to move Mo-Si-B alloys from "lab to fab", i.e. to increase their engineering usefulness, new clean and efficient fabrication method ought to be introduced.

In our project, we propose to investigate a possibility of using **pressure-less Reactive Melt Infiltration (RMI)** approach. For this purpose, we started with experimental studies on interfacial phenomena by using sessile drop method (various T/t conditions), then we moved to small-scale hot dipping experiments. Finally, a dedicated reactor was applied to prepare upscaled samples. Phase structure of produced materials, was investigated by using SEM/EDS, XRD and TEM techniques.

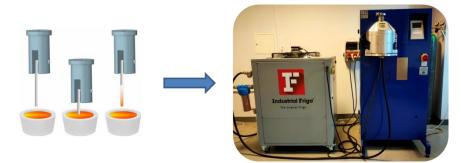


Fig. 1. A scheme of the explored RMI process for a fabrication of Mo-Si-B alloys.

Keywords: Mo-Si-B alloys; Sessile drop method; Liquid assisted processing; Interfaces

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# IMPACT OF HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HIGH ENTROPY ALLOYS BASED ON AICoCuFeNi SYSTEM

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High entropy alloys based on AlCoCuFeNi system were produced by induction meltng with inert gas blow on metal bath surface. For production, where choosen 3 varints of alloys, with chemical composition like in Table 1:

Designation	At. %					
Designation	Al	Co	Cu	Fe	Ni	
D1	7,14	7,14	28,57	28,57	28,57	
D3	13,33	6,66	26,66	26,66	26,66	
D5	20	20	20	20	20	

Table 1. Chemical composition of fabricated alloys

Casting process were conducted into ceramic molds with cylindrical shape, heated to 900°C. After melting, first samples to microstructure, hardness and tensile tests were prepared. Nexly rest of material were divided into pieces and prepared for heat treatment. Based on currently available information's, heat treatment where conducted with scheme in Table 2:

Designation	Type of heat treatment
D1, D3, D5	As cast state
D11, D31, D51	Annealing 1000°C- 5h air cooling
D12, D32, D52	Annealing 1000°C - 10h, air cooling
D13, D33, D53	Annealing 1000°C - 10h, water cooling

Table 2. Type of heat treatment

Microstructure images shows that heat treatment doesn't have significant impact on phase composition and phase distribution (in case of D5 variant where at least 2 types of phases are visible). Comparing hardness result shows that even in case of water cooling variant, significant changes aren't observed. In all variants the highest hardness occur in as cast state variant, what can be a result of impact internal stresses after casting. Results are presented in Table 3:

Hardness HV5								
D1	143	D11	132	D12	130	D13	126	
D3	163	D31	146	D32	126	D33	131	
D5	392	D51	280	D52	281	D53	281	

Table 3. Hardness of tested materials

Tensile test shows many differences and impossible is indication factors which have influence on properties. It was found that heat treatment doesn't affect significantly on properties of this type of materials.

# TELEMEDICAL, WEARABLE CARDIAC SURVEILLANCE SYSTEM BASED ON A TEXTRONIC T-SHIRT

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The increase in the number of cases and diagnoses of coronary heart disease (CHD) in Poland recorded in the second decade of the 21st century is the highest rate among the developed EU countries that joined the European Union before 2000. The presented data show that in Poland in 2017, 1.6 million people suffered from ischemic heart disease, i.e. 4.2% of the entire population and 0.7 percentage points more than the EU average. In the National Health Fund report on health [1], the final conclusions defined two priority actions in regarding CHD, i.e.

• Prevention aimed at reducing disease risk factors,

• Increasing access to early cardiac rehabilitation.

A non-intrusive and easy-to-use system for diagnostics and cardiological supervision for people with cardiovascular diseases was developed, which meets the needs defined above, as part of the doctoral project.

The developed telemedical system processes and analyzes recorded signals and calculated biomedical parameters characteristic of cardiac episodes. As a result of this analysis, feedback can be generated for the patient, indicating the actions he should take to protect himself against a sudden deterioration of his health condition, while simultaneously reporting the episode to the telemedicine surveillance center or to the mobile application of the attending physician. In particular, this will include information warning about the possibility of exceeding the level of supervised biomedical parameters.

The system includes individually dedicated, wearable clothing, equipped with a conductive biomedical signal acquisition network, connected to an electronic registration and wireless transmission module. This module, based on an efficient microcontroller dedicated to mobile applications, will ensure fully autonomous operation, i.e. patient supervision carried out by the system at early warning levels will not require the involvement of a specialist. The tests performed on the model system for recording ECG signals and the respiratory wave concerned adults of different ages, different stature and different physical condition. The tests were carried out on healthy people to exclude any abnormalities in the recording of ECG waves that could result from diagnosed dysfunction of the cardiovascular system or pathology of the conduction system. The purpose of conducting research on many users was to verify the quality of the obtained ECG recordings in accordance with the requirements of medical subject standards.

The developed system can be used in particular in people after a myocardial infarction, when cardiac rehabilitation is necessary, which in the case of the hybrid method shows significant benefits.

[1] National Health Fund on health, Coronary heart disease, Warsaw, 2020

## MATERIAL VALIDATION FOR HEAT EXCHANGERS IN CONCENTRATION SOLAR POWER PLANTS

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Concentrated Solar Power (CSP) represents a promising solution recognized by governments around the world in response to the challenge of climate change. These renewable energies offer a sustainable and renewable alternative to meet the growing global energy demands and reduce greenhouse gas emissions that have experienced an increase since the last century. These systems harness concentrated solar energy to generate electricity by directing solar radiation onto a thermal fluid, which will be transferred to steam to produce electricity through a Rankine cycle. Within the spectrum of CSP technologies, Parabolic Trough (PT) systems offer higher maturity levels.

The relevance of my thesis lies in the optimization of material and heat transfer fluid selection for heat exchangers used in PT systems. This critical component determines the efficiency, durability, and operational safety of CSP plants. My research focuses on evaluating the performance of a wide range of materials in novel molten salts formulations, exploring their suitability to withstand the extreme temperature and corrosion conditions present in CSP environments. Furthermore, the viability of these molten salts as safer and more efficient alternatives to conventional thermal oils used in the industry nowadays is analyzed, thus contributing to the improvement of sustainability and safety in CSP plant operations.

This research not only addresses the immediate need for more reliable and environmentally friendly heat transfer fluids but also lays the groundwork for enhancing the overall performance of CSP systems. By mitigating risks associated with thermal oil usage and introducing innovative solutions, this study aims to facilitate the adoption of CSP technology as a key component of the transition towards a more sustainable future.

The presentation will focus on the relevance of my research for the continuous improvement of CSP technology, specifically in the validation of materials AISI 316L and VM12 under extreme operating conditions of 550°C in long-term test (2000h) in both steam and a low melting point molten salt (HitecXL), allowing for a deeper understanding of their performance in high temperature environments. The justification for this research lies in the need to ensure the proper selection of materials for heat exchangers in CSP plants, which will contribute to improve the efficiency and reliability of these crucial installations for transitioning to a more sustainable energy model in the face of climate change.

## COMPARATIVE ANALYSIS OF 3D PRINTED WALL INSULATION SOLUTIONS FOR ENHANCED ENERGY EFFICIENCY

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This study represents a process to synthesize and critically analyze existing literature on the optimization of 3D printed wall insulation, with an emphasis on the integration of advanced materials such as perlite into sustainable construction practices. Conducted from an analytical perspective, the research meticulously reviews a wide array of published works, spanning peer-reviewed articles, industry reports, and patent documentation, to forge a comprehensive understanding of current methodologies, material innovations, and design strategies within the context of 3D printed construction for enhanced energy efficiency.

The study delves into the complexities of thermal and acoustic insulation challenges in the built environment, scrutinizing the potential of 3D printing technology to revolutionize traditional construction methodology. A critical analysis of material properties, focusing on porous materials like perlite, alongside innovative design configurations, forms the basis of this investigation. The research aims to find key insights regarding the effectiveness of various insulation strategies, particularly in addressing thermal bridging and optimizing energy performance in buildings.

Through a detailed examination of existing studies, this research identifies trends and gaps in the field, highlighting the significant potential of 3D printed walls in achieving superior insulation standards. The analytical journey reveals that strategic layering and structural arrangements of materials, facilitated by the precision and flexibility of 3D printing, can improve thermal and acoustic insulation properties. Moreover, the environmental assessment synthesized from the reviewed literature underscores the sustainability advantages of employing perlite and similar materials, such as reduced carbon emissions and enhanced recyclability.

The study culminates in a set of guidelines for future research and practical applications in sustainable construction, advocating for a comprehensive approach to material selection, design optimization, and environmental consideration in 3D printed buildings. It underscores the necessity for ongoing innovation in material science and architectural design to fully realize the energy efficiency and sustainability potentials of 3D printing in construction.

By offering a critical review on the collective technology surrounding 3D printed wall insulation, this research provides valuable insights for academics, industry professionals, and policymakers alike. It lays the groundwork for future investigations into sustainable building techniques, underscoring the importance of analytical studies in propelling the domain toward practices that are more energy-efficient.

This investigation contributes to scholarly discussions on 3D printed building techniques and insulation methods, establishing a crucial base for upcoming empirical studies. It underscores the revolutionary role of cutting-edge materials and 3D printing methods in creating structures that are both more eco-friendly and energy-saving, leading toward a construction industry innovation that aligns with environmental conservation and innovative practices. This allows for the simultaneous leveraging of the advantages of 3D printing technology and the increase of its market profitability.

The opportunity to conduct this work under the "Doktorat wdrożeniowy 2023" program is greatly appreciated.

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[2] Ayegba, B.O.; Egbe, K.-J.I.; Matin Nazar, A.; Huang, M.; Hariri-Ardebili, M.A. Resource Efficiency and Thermal Comfort of 3D Printable Concrete Building Envelopes Optimized by Performance Enhancing Insulation: A Numerical Study. Energies 15,1069. 2022

[3] I. Hager, A. Golonka, R. Putanowicz, 3D Printing of Buildings and Building Components as the Future of Sustainable Construction?, Procedia Engineering, Volume 151, 2016, Pages 292-299, 2016

#### SELF-ASSEMBLED STRUCTURES FORMED BY NON-IONIC SURFACTANT AND POLYMER

Klaudyna Grzela<sup>1,2</sup>, Katarzyna Haraźna<sup>1</sup>, Kamila Gaweł<sup>1</sup>, Bożena Tyliszczak<sup>3</sup>

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Novel formulations for targeted drug delivery have gained popularity in recent years. Well-designed carriers make it possible to enhance the therapeutic potential of the active substances used, allowing it to be introduced in larger amounts to the target site, This approach may enable the elimination of several side effects associated with oral drug dosage. The self-assembled nanostructures formed of surfactant, polymer and active substance are examples of drug carriers of current interest.

The use of amphiphilic surfactants enables the solubilization of hydrophobic drugs while ensuring their stability and bioactivity [1]. These drug carriers might be used, for instance, for the delivery of anticancer drugs such as cabazitaxel. However, traditional formulations containing cabazitaxel, i.e. Jevtana<sup>®</sup>, contribute to its non-specific dispersion in the body. The other disadvantage of this effective anticancer agent is its poor water solubility. Consequently, in commercially available preparations (Jevtana<sup>®</sup>) it is dosed in combination with non-ionic surfactants and other supportive ingredients [2]. What is important, the literature data showed a significant improvement against prostate cancer cells (C4-2) for cabazitaxel encapsulated in polymeric micelles compared to the drug delivered in the free form [3]. Therefore, the aim of our work is to develop a highly efficient nanoparticle synthesis technology that will enable the delivery of this anticancer agent directly to the desired body's site.

In this work, the selection of methodology for the synthesis of nanoparticles was established. The presented materials were obtained using a non-ionic surfactant, Tween 20, and polyvinylpyrrolidone (PVP) - a polymer approved for use by the United States Food and Drug Administration (FDA). In the first step, the critical micelle concentration (CMC) was determined for Tween 20, PVP and mixtures containing Tween 20 and different concentrations of PVP. For this purpose, a spectrofluorometer employing a molecular probe (pyrene) was used. The size of the particles formed during the synthesis was revealed by the Dynamic Light Scattering (DLS). Then, the zeta potential of the micellar solutions was determined. The zeta potential values indicated the charge that is accumulated on the surface of the nanoparticles. Afterwards, the use of Reichardt's dye and measurements involving a UV-Vis spectrophotometer made it possible to estimate the polarity of the micellar suspensions.

To conclude, the findings show that PVP and Tween 20 can form self-assembled nanostructures that can be used as amphiphilic drug carriers. Due to their solubilization abilities, the carriers constructed in the presented study have the potential to encapsulate hydrophobic drugs.

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## A SYSTEMATIC REVIEW OF CELLULOSE NANOFIBRIL DISPERSION METHODS FOR NANOCOMPOSITES PRODUCTION

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Cellulose nanofibril (CNF) is an abundant and renewable material with excellent intrinsic characteristics such as high strength, insolubility in common solvents, high water uptake capacity, which is interesting for use as reinforcements in composite for many technological applications [1]. However, to effectively promote improvements in the properties of composites, it is essential to ensure the dispersion of CNF in the polymer matrix. Therefore, in this study, cellulose nanofibril dispersion methods were investigated to obtain the composite, analyzing replicability, improvement of properties, environmental friendliness, processing time, complexity, and cost-effectiveness. The PRISMA protocol [2] was conducted to develop a systematic review of scientific articles, considering keywords linked by the Boolean operator ("AND") and criteria presented in Figure 1, resulting in a total of 22 relevant articles.

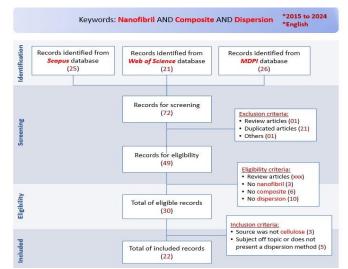


Fig 1. Systematic flowchart of the PRISMA protocol applied in the review.

Several dispersion methods were found including mechanical process (high-intensity homogenization, ultrasonication), physical mixture (plasticizer, water assisted, surfactant, solvent), chemical reactions (TEMPO oxidation, in situ polymerization, enzymatic hydrolysis), thermal treatment or a combination of these, and using different processing mode (extrusion, compression molding, solvent casting). The dispersion method that appears mostly in the research is solvent casting, but it is poorly industrially replicable and uses non-eco-friendly solvents. On the other hand, extrusion is widely used industrially but has limitations related to the dispersion capacity of CNF, indicating the need to develop simple and efficient methods for dispersion and processing CNF composites.

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#### **DIVERSE DIMENSIONS OF CONCRETE 3D PRINTING**

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Additive technologies became increasingly significant as viable means of manufacturing, finding applications in various industrial processes. 3D printing has revolutionized most of the essential base materials fields such as plastics, metals, or ceramics. Advanced structures, strength and topology optimization combined with ease of customization are only a few of the benefits brought along. By overcoming limitations of various additive technologies and materials being used, recent years have witnessed increased interest in three-dimensional concrete printing (3DCP). Importantly, both within the construction industry and among materials science researchers. Significant advancements in solutions for concrete mixtures and methods of its deposition have proven the viability of 3DCP. Despite initial skepticism, 3DCP has emerged as a highly applicable technological extension of traditional methods for building structures. Authors, aligning with this evolution of 3D printing, present various aspects brought by this novel approach. In the presentation, preceded by a brief overview of the technology, attempts to enhance the thermal insulation of structural building walls created by layer-based concrete deposition are discussed. To broaden the perspective, opportunities for reducing material waste and, consequently, process costs compared to traditional construction methods are explored. Additionally, insights on carbon footprint and waste material reduction are presented. Finally, prospects for future developments in the concrete printing field are outlined, with potential areas for deeper exploration, including development trends, scalability challenges, and dynamic 3D printing concrete formulation for enhanced construction performance.

#### SOLIDIFICATION MECHANISM OF DUPLEX POWDER FROM RECYCLED, MIXED 316L/Z100 STEELS

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Recovered metallic waste can be used in additive manufacturing as a feedstock if the subsequent steps of the waste-to-product process are sufficiently mastered. The powder thus obtained could be the answer to the challenges posed by the green energy transition in Europe and the world. However, efficient recycling of stainless steels is a challenge due to the economics of the process and the risk of steel contamination, which can reduce the final corrosion properties of the materials. To address it, we propose a novel approach: the more expensive duplex steel Z100 has been diluted with the cheaper, austenitic steel 316L. Both materials have exceptional corrosion properties at elevated temperatures, but the market price per tonne for Z100 is significantly higher than for 316L. The simultaneous recycling of the 316L/Z100 mixture should result in an economically attractive material with satisfactory corrosion resistance. Before verifying the corrosion resistance of additively formed components, it is necessary to assess the effect of the recycling process on selected final properties of the feedstock in question.

In this study, impact of recycling of Z100 duplex steel mixed with 316L steel on the resulting powders microstructure and chemical composition was investigated. In order to determine whether the characteristics determined were dependent on the size of the powder obtained, analyses were carried out on three separate fractions, differentiated by the volume fraction of the spherical particle diameter within given ranges:  $20 - 50 \mu m$ ,  $50 - 100 \mu m$  and  $125 - 250 \mu m$ . The phase composition analyses carried out reveal the duplex (ferritic-austenitic) character of the powder, depending on the powder fraction studied. At the same time, no differences in chemical composition are found between the different fractions.

The analyses carried out have identified the solidification mechanism of the duplex powder, a scheme of which is shown in **Figure 1** and will be discussed in detail during the conference talk.

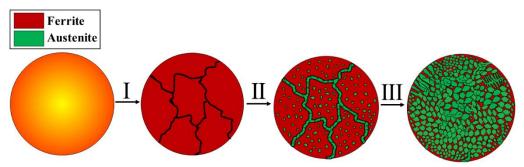


Figure 1. A three-stage scheme of the proposed solidification mechanism.

#### MOBILE DEVICE FOR NON-INVASIVE MEASUREMENT OF THE PULMONARY HYDRATION BY BIOIMPEDANCE SPECTROSCOPY

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The number of heart failure (HF) patients in Poland exceeded 1.24 million in 2022 and is steadily increasing. Hospitalizations resulting from exacerbations of HF, significantly burden the prognosis of patients. Exacerbations are often preceded by a gradual accumulation of fluid in the patient's peripheral and pulmonary tissues [1].

Bioimpedance spectroscopy is a non-invasive body tissue composition measurement. The measurements are made by injecting a weak sinusoidal current through the selected tissue and determining the impedance of the stimulated tissue over a range of frequencies [2].

The aim of the study is to present a prototype of the CardioBIS device and dedicated algorithm for the analysis of bioimpedance spectroscopy, impedance cardiography and ECG signals to support the process of diagnosis and early detection of cardiac failure by detecting the growing accumulation of fluids in lungs.

The CardioBIS device was created as part of the internal project CardioBIS – Multiparameter cardiac telemonitoring system by Łukasiewicz Research Network – Krakow Institute of Technology, in the Centre for Biomedical Engineering (Fig.1). The CardioBIS device is designed for the non-invasive recording of bioimpedance in the range of 5 kHz to 750 kHz and electrocardiographic signals from electrodes attached to the patient's body. The developed device integrates a single frequency impedance cardiography for continuous monitoring of hemodynamic parameters and a multifrequency impedance spectroscopy for periodic hydration measurements.

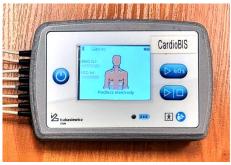


Fig.1 The CardioBIS device. Own source.

Conducted experiment allowed to compare results obtained from human and fixed resistor, with value approximately corresponding to the human impedance at low frequencies. Based on these, an electronic model was then designed. The observed signal waveforms confirmed the compatibility of the adopted electronic model.

Further work will focus on a development of the signal analysis algorithm for determining hemodynamic parameters from impedance cardiography and ECG signals and predicting the occurrence of HF exacerbation through periodic monitoring of lung hydration.

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# RELATIONSHIP BETWEEN MANUFACURING METHOD AND MICROSTRUCTURE WITH SELECTED MECHANICAL PROPERTIES ON CEMENTED CARBIDES

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Cemented carbides are one of the most important tooling materials in modern machining. Their properties connected with high hardness and withstanding high temperatures during working make them suitable for shaping materials into desired dimensions and roughness. Nowadays, industry looks for new possibilities, i.e. using alloys with various elements percentage in chemical compositions, manufacturing more tools from ceramics or other types of carbides (i.e. based on TiC), inventing new methods of sintering, or applying coatings.

Spark Plasma Sintering method is an unconventional way to manufacture such carbides. The difference between conventional methods and SPS are reducing sintering time up to several minutes and lowering the temperature of the process (up to 30%) [1]. The main principle of SPS method is to use high-temperature plasma in intervals lasting dozens of seconds. Because of this, it can be characterized by low voltage but high current. Current directly heats the powder.

Tests were conducted on selected cemented carbide grades S30 and S40S with well known chemical compositions. Samples were bought on the market and sintered by SPS method. It allowed to obtain comparable elements in terms of laboratory tests. Parameters as pressure and time for sintering process were constant, and the variable was temperature. Process was done on SPS HP 5 apparatus.

Laboratory tests consisted of measuring friction coefficient, cross-section area of traces and deriving wear factor, following by SEM images of obtained traces for proving earlier results. For the microstructure light optical microscopy was used to: figure out existence of pores or grain expansion, checking size of grains, the distribution of carbide grains in cobalt matrix, and checking the surface for any impurities. Tomography images were done to check if the samples are homogeneous and if there was an existence of any cracks. Vickers' microhardness was measured to prove that unconventional sintering method had an impact on it.

Results showed that SPS method indeed had an influence on the microstructure and mechanical properties of samples. Friction coefficients was lowered. Cross-sections from profilometer and derived wear volume showed that for S30 this parameter was up to three times lower, for S40S the difference was up to ten times. SEM images for S30 samples showed that the traces were shallow, and any lacks could be classified as delamination or micromachining. Optical microscopy showed that sintered samples had smaller grains. Tomography images showed that samples were homogeneous and there were not visible cracks. Microhardness tests showed that sintered elements had higher values in Vickers' scale.

Given the circumstances, it was shown that SPS method had an influence on structure and properties of cemented carbides. During the process even the slight change in temperature can affect on results. Depends on the grade and its application, the alternative method of sintering is more or less suitable.

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Acknowledges: Sintering service and materials for testing were funded by the Project Based Learning, in the IX competition under the program Inicjatywa Doskonałości – Uczelnia Badawcza, Silesian University of Technology, Gliwice.

#### ANALYSIS OF PLASMA SPRAYED CO-BASED ALLOY WITH HIGH-GLASS FORMING ABILITY

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Amorphous metals have been of interest for researchers since the 1960s of the twentieth century. Since then, alloys based on different chemical elements with high glass-forming ability have been discovered. Typical values of the critical cooling rate to the developed amorphous structure for these alloys range from  $10^2$  to  $10^4$  K/s. The technology used for developing amorphous metals includes casting methods like melt spinning- into ribbons, die casting- into simple shapes like plates, rods, or disks; atomization for obtaining powder, and thermal spraying like high velocity oxygen fuel or cold spraying for obtaining coatings. One of the methods that only several researchers have conducted for Co-based alloys with glass forming ability is plasma spraying. Plasm spraying uses the directed plasm jet to heat and accelerate the material particles, which then hits the substrate and forms coating.

In this work the atmospheric plasma spraying (APS) was used to produce the coating from not previously tested the alloy Co<sub>47.6</sub>B<sub>21.9</sub>Fe<sub>20.4</sub>Si<sub>5.1</sub>Nb<sub>5</sub> with high glass formation ability. As an aim, the optimal parameters in the shooting distance criterion were analyzed. The coatings were sprayed in powder size from 50 to 80 µm. The powder was supplied radially with a feed rate of 15 g/min. As a plasmatic gas an argon was used, the flow was set at 8 slpm. Plasma power was 22 kW. The parameter that was changing to establish the optimal parameter was the spraying distance 90, 100 and 110 mm. After the coatings were sprayed, the structure was tested by scanning electron microscopy (SEM) analysis and X-ray diffractogram analysis, the surface roughness was measured by a profilometer, and the image of it was captured by SEM. Porosity was estimated by graphic analysis of SEM images.

As was expected, the smallest spraying distance (90 mm) allowed to obtain the least porous structure of coatings. Structure analyzes showed local delamination in all coatings, but the highest amount was observed for the coatings developed with a spraying distance of 110 mm. In addition, in those coatings the highest amount of partially deformed particles was observed. The roughness of all samples was similar. The SEM analysis with material contrast and X-ray diffraction analysis show absence of a second phase in any coatings. This observation leads to the conclusion that the atmospheric plasm spraying used is a proper technology for developing metallic glass coatings from a  $Co_{47.6}B_{21.9}Fe_{20.4}Si_{5.1}Nb_5$ . As the optimal spraying distance from this work is 90 mm, this distance ensures high enough energy to develop good density coatings and in combination with other parameters ensure 100% X-ray amorphous structure of those coatings.

#### A NEW METHOD FOR MEASURING OPEN POROSITY

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Roughness parameters related with material ratio curve are atypical. They are calculated differently than the most commonly used height parameters of roughness [1]. There are studies in the literature that analyze volume parameters in order to obtain additional information related to the analysis of open porosity [2-3].

The aim of the studies was to develop a new method for measuring open porosity using surface texture analysis.

Investigations were carried out in two stages. For roughness analysis confocal profiling method was used. In order to determine the open porosity, an analysis of selected volume parameters was performed. First tests of the method were performed with use surface roughness standard, which contains cross grating. This standard may represent a sample with known and defined open porosity. After preliminary calibrations of the method the three real porous sintered materials were prepared using Selective Laser Sintering method. The porosity of the sintered samples was tested using image analysis (grid method), which was the reference for the results of the surface geometrical structure measurements. The obtained results regarding open porosity are consistent with those achieved using the selected reference method.

The main advantage of proposed method is the shortening of the analysis time of samples. Results from a single device (profilometer) provide information on both the surface roughness and open porosity of the sample.

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## COMPARISON OF THE CONTROL SYSTEMS OF THE ARM-100 AND ARM-200 REHABILITATION ROBOT

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Limb disability resulting from lifestyle diseases or injuries impairs the performance of everyday activities and limits work opportunities. In practice, the treatment of such conditions involves rehabilitation that requires tedious repetition of recommended movements of the impaired limb with the participation of an experienced rehabilitator. Rehabilitation exercises using a robot can, first of all, shorten the time from the onset of disability to the start of the rehabilitation process. Research proves that starting rehabilitation quickly is a key factor in effective rehabilitation [1]. The use of robots in the rehabilitation process allows the rehabilitator to be relieved of the burden of performing physical work and also allows for the rehabilitation of several patients at the same time by one rehabilitator. An additional value of using robotic systems is the ability to measure the exercises performed by the robot's sensors. This allows to track the progress of rehabilitation, which may additionally motivate the patient to continue working, which is particularly important in the case of many months of tiring exercises.

The work presents developed control system for a rehabilitation robot and comparison shown the capabilities of the control systems of prototype rehabilitation robots ARM-100 and ARM-200, which were created at the Institute of Medical Technology and Equipment, currently the Łukasiewicz Research Network - Krakow Institute of Technology.

The control system for the new ARM-200 robot has been developed from scratch, using currently available components and slightly changing the philosophy of the adopted solution. The main assumption of the designed system was to develop robot node controllers that can be placed very close to the controlled drive to minimize the number of cables passing through the moving robot nodes. The change in philosophy was to define the functionalities of the robot node, which are not present in all nodes, and which can be implemented as an optional modules. The functions that have been removed from the basic package include: elements enabling control of the extension actuator and brake control. It allows us to limit occupied space by the basic module. To build the new controller, we use a new 32-bit microcontroller based on the Cortex M4 core with floating point unit and clocked at 120MHz, previously we use 8-bit microcontroller with a clock speed of 40MHz. This change allowed the microcontroller to be loaded with new tasks that improved control quality and safety. One of the new functions is the ability to automatically reconfigure the robot from right to left limb and vice versa. An autonomous mechanism for each node to track other nodes was also added, which was used to "stiffen" selected nodes in certain limb positions. A mechanism for automatic addressing of robot nodes has also been added, which may be important during future servicing of the robot.

Due to the complexity of the topic, work will be carried out in the future to improve many elements of the developed system. Additionally, work is currently underway to create a 3D environment for training with a robot in the form of rehabilitation games.

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# PARAMETERS OF THE PROGRAMMABLE PULSE GENERATOR DEDICATED TO IRREVERSIBLE ELECTROPORATION IN ONCOLOGY THERAPY

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Cancer, despite intensive progress in medicine over recent years, is still one of the most common causes of death. One of the methods of therapy used in the treatment of cancerous tumours is ablation. Nowadays, thermal ablation is most often used. Thermal ablation causes tissue necrosis. An alternative to thermal ablation methods is non-thermal ablation using the irreversible electroporation method. It involves affecting tissues with a series of short high-voltage pulses, causing permeabilization of cell membranes [1]. Permeabilization of cell membranes results in the process of apoptosis, which is the natural death. Ablation by irreversible electroporation does not cause tissue necrosis. Irreversible electroporation is safe for adjacent tissues such as nerves or blood vessels because it does not damage them. Cells that die as a result of irreversible electroporation are then removed from the body through natural processes in living organisms.

A device called an electroporator is used to generate programmable microsecond high-voltage pulses used for electroporation. The main module of the electroporator is a generator of a series of high-voltage pulses with programmable amplitude and time parameters within wide range. The appropriate selection of the range of programmable parameters is important due to the expected use of the electroporator being developed for the future research on the use of irreversible electroporation for the ablation of various cancer tissues, first using animal models, and then conducting clinical research on humans. The paper presents the assumptions made, based on the analysis of available literature on the subject, regarding:

- shape of pulses,
- regulation range of the amplitude and duration of a single pulse,
- regulation range of the number and repetition time of pulses in a packet,
- regulation range of the number and repetition frequency of pulses packets.

Based on the approved assumptions, will be carry out research to develop a programmable high-voltage pulse generator for use in an electroporator.

This work was supported by the resources of the Łukasiewicz Network Centre, Poland, within the Project no 3/Ł-ITAM/CŁ/2021.

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## APPLICATORS FOR TISSUE ABLATION BY IRREVERSIBLE ELECTROPORATION METHOD IN ONCOLOGY APPLICATIONS

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One of the newest ways of fighting with cancer disease is a tissue ablation using irreversible electroporation [1]. As a result of the impact on tissues using short high-voltage pulses, irreversible permeabilization of cell membranes occurs. This, in turn, leads to the process of apoptosis, which is a natural, controlled process of cell death in a multicellular organism. Non-thermal ablation by irreversible electroporation may be more effective and safer than thermal ablation, which is currently widely used for therapeutic tissue destruction in medicine. Electroporation does not cause tissue necrosis and is safe for neighboring structures, such as blood vessels and nerves.

To generate microsecond pulses of high voltages, it is necessary to develop a device called an electroporator. In addition to the high-voltage pulse generator, conducting non-thermal ablation also requires applicators through which the pulse will be delivered to the tissue. The paper presents the concept of a prototype of an innovative applicator [Fig. 1] for tissue ablation using irreversible electroporation, which was developed on the basis of a literature review. Appropriate materials were selected for both the electrode core and for the conducting and insulating coatings of the surface. The initial design of the applicator was modeled, specifically the shape of the electrode and the insulated handle, which must be safe for use. Currently, research is being carried out on a permanent and safe connection of the wire with the electrode and a safe device connection, which in the future will enable the connection of the applicator to the electroporator.

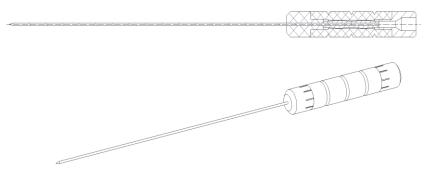


Fig 1. The concept of a prototype of an innovative applicator

The development of an electroporator with applicators will allow preclinical research on animal models and clinical trials on patients, confirming the effectiveness of therapy using electroporation ablation. These studies will aim to optimize therapeutic processes in oncological applications.

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## EXPLORING THE MECHANICAL AND THERMAL PROPERTIES OF BIOCOMPATIBLE Zr – Cu – Ag METALLIC GLASSES

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The study focused on examining the mechanical properties of metallic glasses derived from the Zr-Cu-Ag system, highlighting their advantages over crystalline counterparts, particularly in terms of biocompatibility and mechanical performance. Two specific alloys,  $Zr_{50}Cu_{37}Ag_{13}$  and  $Zr_{42}Cu_{40}Ag_{18}$ , were selected for investigation due to their proximity to the triple eutectic points, which typically ensures good glass-forming ability.

The alloys were fabricated using high purity elements and cast via suction casting. Chemical composition analysis was conducted using X-ray fluorescence spectroscopy (XRF) and X-ray microanalysis (SEM-EDS). The amorphous nature of the alloys was confirmed through scanning electron microscope (SEM-BSE) and X-ray diffraction (XRD). Oxygen content, an important factor in metallic glass properties, was determined using the inert gas fusion method.

Mechanical properties, including hardness and behavior under compression at different strain rates, were evaluated using Vickers hardness testing and uniaxial compression tests. Fracture analysis via scanning electron microscopy (SEM-SE) revealed differences in fracture behavior between alloys with crystalline phases and fully amorphous ones, with the latter exhibiting plastic deformation.

Additionally, differential thermal analysis (DTA) was employed to assess glass transition behavior, crystallization, and melting temperatures, providing insights into the alloys' glass transition capacity.

Overall, the study concluded that  $Zr_{50}Cu_{37}Ag_{13}$  and  $Zr_{42}Cu_{40}Ag_{18}$  metallic glasses possess promising characteristics for bioengineering applications due to their high strength, moderate glass-forming ability, and absence of toxic elements in their composition. These findings suggest potential applications in fields where biocompatible and mechanically robust materials are required.

# EXPLORING THE IMPACT OF ALUMINIUM POWDER AND HYDROGEN PEROXIDE ON FOAMED CONCRETE BEHAVIOR IN 3D PRINTING APPLICATIONS

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Foam concrete, known for its lightness and insulating properties, shows promise in construction applications, especially in the context of additive manufacturing. In this study, by adding aluminum powder and hydrogen peroxide to the concrete mix, the resulting effect on the material's foaming properties, mechanical properties and its printability were analyzed. The presentation will discuss experimental methodologies, including formulation of concrete mixtures and printing parameters, as well as analysis of foam behavior and the mechanical strength of the material. During the tests, the addition of aluminum powder had a significant impact on increasing the compressive strength only for concrete samples with the addition of silica fly ash. For concrete samples based on Portland cement clinker and post-production dust, the addition of aluminum powder and hydrogen peroxide (H2O2) significantly reduced this value. Additionally, for both types of concrete, the addition of hydrogen peroxide resulted in a significant reduction in average bending strength. Additionally, the implications of these findings for advancing the use of foamed concrete in 3D printing technologies will be outlined, offering insight into optimizing material formulations to increase performance and applicability in construction projects.

#### PREPARATION OF HYDROGEL MATERIALS USING A BIO-CROSSLINKING AGENT

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Hydrogels are materials with a three-dimensional structure obtained by cross-linking of synthetic or natural polymers. Polymers of a natural origin are becoming increasingly used as raw materials to prepare the hydrogels. The most frequently used groups of natural polymers are polysaccharides (alginate, chitosan, hyaluronic acid) and proteins (collagen, gelatin, fibrin). The medical and pharmaceutical applications of hydrogels include wound healing dressings, controlled drug delivery systems, and soft tissue engineering. Currently, material engineering is primarily based on synthetic cross-linking agents, such as N,N'-methylenebisacrylamide, ethylene glycol dimethacrylate, epichlorohydrin, and glutaraldehyde. The use of this type of compounds is associated with the risk of toxic effects due to insufficient purification of the material. An alternative may be the use of natural cross-linking agents that have many benefits from using, such as no toxic effect, or no need for purification process [1-4].

The research was aimed at obtaining polymer materials cross-linked with a plant-derived cross-linking agent.

A series of natural cross-linking agents were obtained using modern extraction methods supported by ultrasounds and microwaves. The extracts were characterized by determining the composition of the mixture using high-performance liquid chromatography and antioxidant properties using the Folin-Ciocalteu method and the DPPH method. Then, the obtained mixtures were used as a cross-linking agents during the cross-linking reactions of the selected natural polymer, which was gelatin. The obtained hydrogel materials were subjected to physico-chemical analysis (cross-linking degree, swelling degree), structural analysis (Fourier-transform infrared spectroscopy) and morphological analysis (Scanning electron microscopy).

Based on the conducted research, an improvement in the physico-chemical properties of hydrogel materials containing a cross-linking agent was found. Moreover, FT-IR analysis of these materials allowed to observe shifts in the absorption bands characteristic of gelatin, which may indicate the occurrence of chemical cross-linking reactions of polymeric films. SEM micrographs confirmed the change in the cross-sectional morphology of the obtained samples as a result of the addition of a cross-linking agent.

The conducted research allowed to confirm the positive impact of the developed bio-crosslinking agent on the basic properties of polymer materials.

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#### USE OF WASTE MATERIALS IN THE PRODUCTION OF ALKALI-ACTIVATED BINDERS

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This study investigated the possibility of synthesizing alkali-activated binders based on waste materials of local origin. For this purpose, materials such as amphibolite (A), fly ash from lignite combustion from Belchatow (PB), chalcedonite (Ch) and diatomite dust (D) calcined at 900 °C were used. Metakaolin (M) was an additional binder modifier. The waste materials were examined by methods such as laser particle size analysis, X-ray fluorescence analysis (XRF), X-ray diffractometry (XRD), and scanning electron microscopy analysis (SEM). The reaction activator was a 10M solution of NaOH and water glass in a weight ratio 1:1.5. Mixtures of alkaliactivated base materials were heated for 24 hours at 60 °C and then conditioned for 28 days under laboratory conditions. The binders produced were microstructurally analyzed (SEM) and subjected to flexural and compressive strength tests. The highest compressive strength results were obtained for samples based on chalcedonite, diatomite, amphibolite and metakaolin - around 20 MPa. Flexural strength tests showed that the highest results were obtained by binders based on diatomite, amphibolite, fly ash from Belchatow, and metakaolin - over 12 MPa, while the lowest values were obtained by binders based on chalcedonite, diatomite, amphibolite and metakaolin - about 4.8 MPa.

#### NUMERICAL ANALYSIS (FEA) OF UPPER LIMB REHABILITATION ROBOT CONSTRUCTION

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One of the most significant health problems these days is locomotor dysfunction, which is a consequence of aging of society, injuries and diseases. Returning to full mobility often needs long-term and tedious rehabilitation process, access to which is usually limited due to the insufficient number of therapists. Rehabilitation robots are now increasingly used to increase the availability and improve the efficiency of the rehabilitation. This shortens its duration, reduces the therapist's involvement in an individual patient rehabilitation, as well as enables an objective quantitative and qualitative assessment of its progress [1].

Considering that robots used in rehabilitation process are treated as medical devices, they must meet a number of requirements. Meeting the determined criteria is intended to ensure the reliability of performance and, most importantly, safe use for patients and therapists. Therefore, numerical analysis is used in the designing process to predict the behaviour of the device under defined loads and operating conditions. This enables to check if the designed model and its properties are relevant. This approach makes it possible to eliminate many design problems before producing the physical version, consequently saving time and money.

This work presents one stage of design process of the upper limb rehabilitation robot construction. This procedure involved preliminary numerical analysis based on Finite Element Method, using Ansys Workbench software. Conducted research comprised material features selection and strength analysis of particular construction elements of the exoskeleton. The analysis was conducted for two construction forms, whose 3D models were created in Autodesk Inventor environment. Preprocessing phase was described, involving a preparing the model for analysis, its simplification, determining the assumptions and boundary conditions, as well as correct meshing of the model. Selection of the material features was based on results which were visualised as equivalent stress and displacement maps for three material variants in two setting variants of the exoskeleton. Optimal material variants was thoroughly analysed in terms of stress and displacement states.

The research confirmed that by applying Finite Element Method analysis, many design problems can be eliminated without making the physical version of the device. Strength analysis enabled to locate "weak points" of the construction, which could have been removed during designing phase. This approach is certainly reasonable, as it saves time and money, and also helps to predict the device behaviour in extreme conditions. It enables to verify whether the device is able to withstand the defined forces and loads in every analysed case, which consequently has to ensure safety of use. It should be noted that the stage of model preparation and making assumptions for the strength analysis may seem relatively simple, but in fact it requires a lot of knowledge and experience, and it has the greatest impact on the results obtained.

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#### INTEGRATED DESIGN APPROACH FOR HIGH ENTROPY ALLOY DEVELOPMENT

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High-entropy alloys (HEAs) with their vast compositional space offer exciting avenues for designing materials with exceptional properties for diverse applications. Predicting the stable phases and performance of these intricate alloys within this landscape remains a significant challenge [1]. This work proposes an interdependent approach that integrates the strengths of computational tools, fundamental principles, and consistent validation to optimize HEA design for enhanced performance [2].

This approach leverages the strengths of both worlds. Computational tools offer efficient initial exploration of the compositional landscape, identifying promising candidate compositions [2]. However, relying solely on these tools can miss crucial nuances. This is where fundamental principles like the Hume-Rothery rules, valence electron concentration (VEC) analysis, and understanding atomic size mismatches offer deeper insights into phase stability based on chemical and physical interactions between elements, refining computational predictions and guiding targeted exploration [3]. Finally, iterative design and validation close the loop by experimentally validating predicted phases and incorporating this feedback to inform and refine the computational models [4].

This hybrid approach offers several advantages. Firstly, it enables more accurate phase prediction, reducing the risk of unsuccessful experiments and accelerating the design process. Secondly, understanding the interplay between composition, phases, and fundamental principles allows for tailored material properties. The effectiveness of this approach is evident in various HEAs with outstanding properties. The NbTaMoW refractory HEA exhibits exceptional high-temperature strength, surpassing conventional superalloys, thanks to a design process that combined computational calculations with VEC analysis and atomic size mismatch considerations. Furthermore, the FeCoNiCrMnCu HEA possesses remarkable shape-memory properties, achieved through a process combining DFT calculations with experimental validation to tailor the composition for desired transformations [5].

By integrating computational power with fundamental knowledge and iterative validation, this synergistic approach unlocks the full potential of HEAs and paves the way for developing next-generation materials with exceptional properties, revolutionizing various industries.

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#### ULTRASONIC CASTING OF Zn AND Zn - Bi ALLOY

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The gravity casting is one of the older method of alloy production, with no high requirements of advanced technology. Although gravity casting is relatively easy to proceed, a problem with porosity, size participations and distribution of participations appears. One of the method used to limits the listed problems is use of ultrasonic in process of casting.

It was discovered that with using of the ultrasonic, casted mould characterized in higher mechanical properties without addiction of other elements in alloy[1 - 5]. In current studies the authors examined the influence of the ultrasonic at Zn – Bi 2% alloy. The research reviles the sufficient influence of ultrasonic at the crystallization process. In case of both ingots (Zn and Zn-2%Bi) the differences in microstructure and mechanical properties could be observed. The methodology was based on casting two sample from the same ingot with identical configuration. One of the samples was used as the reference (casted without ultrasonic) and the other one to examine ultrasonic influence. The ultrasonic casting was proceeded with use of sonotrode with frequency of 35 kHz located at the underside of designed crucible. Obtained samples has a shape of circle with a diameter of 50 mm and thickness of 10 mm. Next, the samples were conducted a series of examination tests including tensile strength test, Vickers hardness test, microscope examination. Tests of ultrasonic ingots showed enhanced organization of microstructure as well as the fragmentation of a grains. It was vividly seen especially in case of bismuth phases for Zn-2%Bi alloys.

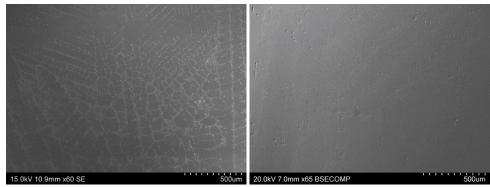


Fig.1 The microstructure of Zn – Bi 2% samples; without ultrasonic (left side), with ultrasonic (right side).

Although hardening coefficient calculated in range of plastic flow for the ultrasonic samples raised up, the basic mechanical properties ( $Re_{0.2}$ ) stood at the same level in both group. Last, but not least, the down fall of statistic error (standard deviation) during microhardness tests was noted.

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